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1. Your reference
LRD-GB-2-429

2. Patent application number *(The Patent Office will fill in this part)*
0304356.9
26 FEB 2003

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*
K.U.Leuven Research and Development - Groot Begijnhof 59 - 3000 Leuven
Represented by Dr. Patrick Chaltin, IPR Officer
Patents ADP number *(if you know it)*
7665649003

If the applicant is a corporate body, give the country/state of its incorporation
Belgium

4. Title of the invention
New emulsification technology

5. Name of your agent *(if you have one)*
K.U.Leuven R&D
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7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application	Number of earlier application	Date of filing *(day / month / year)*

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Any other documents *(please specify)*	1 fax cover sheet 1 fee sheet with request for fax back service

11. I/We request the grant of a patent on the basis of this application.

Dr. Patrick Chaltin - IPR Officer — Signature : — Date 26/02/2003

12. Name and daytime telephone number of person to contact in the United Kingdom: Hubert Velge, +44 7940 540 397

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NEW EMULSIFICATION TECHNOLOGY

Field of the invention

The invention relates to a new method and related equipment for the preparation of emulsions. According to the method of the invention stable emulsions are obtained by conducting an unstable pre-mixture of immiscible fluids through a magnetic field.

Background of the invention

Emulsions are broadly defined as metastable colloidal dispersions of liquid droplets in another liquid phase. The major emulsions include, oil-in-water and water-in-oil emulsions, benzene, toluene, zyline and nitro-benzene based emulsions.

Besides two liquid phases and an emulsifier, the formation of an emulsion takes energy and occurs at conditions far from equilibrium. In general, the formation of an emulsion comprises two steps. In an initial step the two liquid phases are mixed in the presence of a suitable amount of emulsifier in order to create droplets of the dispersed phase in the continuous phase. Thereafter, the droplets are disrupted by shear forces or by local pressure differences, i.e. by inertial forces, resulting in a more stable emulsion. At present different types of mechanical emulsification processes are used in the production of finely dispersed emulsions, each process requiring specific equipment. Within the mechanical emulsification systems four major categories can be discriminated: droplet disruption in high shear rotor-stator systems, droplet disruption by ultrasound, droplet disruption in high-pressure systems and droplet formation at micropores (microporous membranes or microchannels). Also non-mechanical processes are applied such as precipitation of the dispersed phase previously dissolved in the continuous phase, phase inversion and the phase inversion temperature method.

Emulsions are either produced as consumer products or for applications within a broad range of industries. The areas, in which emulsions are used, include, in a far from exclusive list, foods, paints, cosmetics, pharmaceuticals, explosives, rocket fuel, etc. Most applications or products require that emulsions have maximal storage stability. The storage stability refers to the period that the emulsion can be kept before it separates into different phases. The five mechanisms, that can be identified in the process of breaking down an emulsion, are Ostwald ripening, creaming, aggregation, coalescence, and partial coalescence. There are two ways in which the process of breakdown of an emulsion can be influenced: the use of mechanical devices to

control the size of the dispersed droplets and the addition of stabilising chemical ad (emulsifiers) to keep the emulsion dispersed.
The present invention describes a new physical method to produce finely dispersed stable emulsions, using magnetic fluid treatment devices.

SUMMARY OF THE INVENTION

In a first object the invention relates to a method for the preparation of an emulsion by conducting an unstable mixture of immiscible fluids through a magnetic field. In a more preferred embodiment the mixture further comprises one or more emulsifier.
In a second object the method relates to a device for the preparation of an emulsion according to the method of the invention. Said emulsification device comprising a magnetic fluid conditioner mounted in a circuit comprising a liquid containing portion, whereby the fluids contained in the liquid containing portion can be conducted through the magnetic field of the magnetic fluid conditioner. Optionally, a mixer is mounted on the liquid containing portion. In a more preferred embodiment the device comprises a pump, which allows controlling the speed at which the fluids are conducted through the magnet. In a more preferred embodiment the fluids are recirculated through the magnet back into the liquid containing portion. In an even more preferred embodiment the re-introduction of the magnetically treated mixture into the liquid containing portion is done with proper care in order to avoid that the magnetically treated mixture mixes with the liquids contained in the liquid containing portion. Optionally, the pre-mixture is conducted through a magnetic field when transferring the pre-mixture to the liquid containing portion.
A third object of the invention is the storage of an emulsion in the liquid containing portion of the emulsification device of the present invention. Intermittent conduction of the emulsion through the magnetic field may allow prolonging the storage stability of the emulsion.
A fourth object of the invention relates to altering the characteristics of an existing emulsion (such as the micelle size) by conducting said emulsion through a magnetic field.

DETAILED DESCRIPTION OF THE INVENTION

List of figures

Figure 1: Representation of different configurations of the emulsification device.

A. Emulsification device used in Example 1. The device comprises a liquid container consisting of a Haake bath wherein a centrifugal pump is submerged. Said pump allows conducting the liquid content of the Haake bath through the magnetic device back into the bath.

B. Emulsification device used in Example 2 and example 4: a liquid reservoir consisting of a cylindrical flask having a conical bottom is equipped with a loop containing a peristaltic pump and a magnetic device . The magnetic device is positioned at the inlet of the reservoir.

C. Emulsification device used in Example 3: a liquid reservoir consisting of a cylindrical flask having a conical bottom is equipped with a loop containing a peristaltic pump and a magnetic device. The magnetic device is positioned at the outlet of the reservoir.

D. Emulsification device used in Example 5: a liquid reservoir consisting of a cylindrical flask having a conical bottom is equipped with a loop containing a peristaltic pump and a magnetic device. The magnetic device is positioned at the outlet of the reservoir. The cylindrical flask, the magnetic device and part of the tubing is submerged in a water bath in order to cool the circulating emulsion.

Figure 2: The sizes of the fat globules in semi-skimmed milk after 5 and 10 minutes of magnetic treatment, compared to untreated milk. Because measurement is executed assuming the viscosity of the samples to be equal to water, the dimensions are relative instead of absolute.

Figure 3: The size distribution of the lipid micelles in whole milk after 5, 10 and 30 minutes of magnetic treatment, compared to untreated milk. Because measurement is executed assuming the viscosity of the samples to be equal to water, the dimensions are relative instead of absolute.

Description

The present invention describes the surprising finding that the passage of an unstable mixture of immiscible liquids through a magnetic field results in a stable emulsion. Furthermore, different operational parameters were identified to be relevant to the preparation of a stable emulsion by means of a magnetic device. The influence of some of these parameters is illustrated in the Examples.

The equipment used in the present study comprises a magnetic device, such as the devices currently used in magnetic water conditioning to prevent scale. The magnetic device was mounted in a circuit containing a pump and a reservoir (Figure 1). The emulsion is prepared by pre-mixing the oil, water and surfactant before filling it in the reservoir. Thereafter, a stable emulsion was obtained by circulating the premix in the unit. The emulsion stability was evaluated by observing the spontaneous macroscopic phase separation by eye after a period of time. In a series of experiments it was demonstrated that following parameters were affecting the storage stability of the obtained emulsion:

1. Influence of flow rate of mixture through the magnetic field

The stability of the obtained emulsion is dependent on the linear flow rate of the mixture through the magnetic field. In each particular application and equipment, there is a threshold flow, below which there is no improvement of the stability of the pre-mixture. The gain in emulsion stability increases with the linear flow rate. The transition from laminar to turbulent flow, and the occurrence of undesired cavitation impose the maximum linear flow rate. In each application and equipment, an optimum flow rate exists.

2. Number of passes through the magnetic field

The stability of the emulsion is improved when the number of passages through the magnetic device is raised to a maximum effect. The improvement of the emulsion stability is most significant in the first passes. Prolonged circulation has no adverse effect. Below the threshold flow, raising the number of passes does not help to improve the stability of the emulsion.

3. Time lapse between passes

The time lapse between two passes through the magnet while the mixture is in the liquid containing portion or tubing is another parameter of importance to the stability of the emulsion. In a preferred system, the pre-emulsion is treated a number of times with short time intervals as to reach sufficient stability for storage.

4. The emulsion storage reservoir

The nature of the reservoir is an important parameter for several reasons.

The shape of the reservoir is important to ensure that the entire volume of the emulsion is treated and to avoid zones where the premix stagnates. This would increase the time lapse between treatments for part of the volume. The stagnant zones comprising an emulsion/premix with larger micelles can be responsible for coagulation and phase separation. Experiments with differently shaped reservoirs illustrate the importance of this parameter. Another point of concern is the mixing of the treated emulsion with the stored volume. Mixing is to be minimised in order not to increase the lapse time.

5. Alternative magnetic devices

Most of the experiments were performed with a device manufactured by the CEPI-co company (Borgerhout, Antwerp). (CEPI® R1/2D). This device contains two AlNiCo magnetic bodies with a specific shape as to create two consecutive orthogonal magnetic fields inside the fluid passing through. Some experiments were executed with different types of magnetic devices. Two clamp-on type devices, having the magnets positioned outside of the fluid, were used. One of these clamp-on units had a FeNdB composition, the other one AlNiCo. The stability of the premix was improved in experiments with these alternative magnetic devices as well.

EXAMPLES

EXAMPLE 1: Magnetic preparation of an oil-in-water emulsion using a Haake circulation bath as liquid-containing reservoir

<u>Research equipment and procedures</u>

1. Preparation of the premix

0,930 kg of fatty acid and 0,061 kg of surfactant were added to 2,001 kg of de-ionised water. The fatty acid 'radiacid 0166' and the surfactant 'radiasurf 7403' were provided by Oleon company. This mixture was vigorously stirred with an IKA RE16 mixer at 300 rpm. The suspension was slowly heated to a temperature of 60-65°C with an IKA RCT heater. The temperature was controlled with an IKA ETS-D4 fuzzy-apparatus.

After 1 hour the temperature had reached 60,7°C. 11,7 g of a 50 wt% NaOH-solution was added dropwise, while stirring of the mixture was continued. In doing this, the temperature increased to 62,2 °C. As a result, a white suspension was obtained.

2. Magnetic Treatment

The hot suspension was poured into a Haake circulation bath, while mechanical stirring is immediately started (IKA RE16 mixer at 200 rpm). The suspension was pumped at a rate of approximately 3L/min through 2 commercial CEPI®-magnets (CEPI® R1/2D) in series. The experimental configuration corresponded to the scheme in Fig.1A. The treatment was continued for 6 hours, while the suspension was allowed to cool spontaneously.

3. Sampling

The temperature of the mixture was measured during the treatment. At certain moments a sample of the suspension was taken and stored at room temperature for visual inspection. An overview of the sampling data is given in the Table 1 .

time (min)	temperature (°C)	sample
0	58,8	s0
15	51,6	s1
60	39,9	s2
180	30,6	s3
360	28,8	s4

Table 1: timing of sampling and sample notation

4. Determination of emulsion stability

Samples were collected in small glass vials (7 ml). The phase separation of the emulsion in the vials was observed by eye.

Results

Sample s0 was separating quickly into a white yellow top layer and a white water layer. The emulsions s1-4 appeared by eye to be stable after 6 hours, even the sample taken after 15

minutes. After overnight storage at room temperature most of the samples showed a phase separation, though a better stability was observed the longer the magnetic treatment was continued. None of the applied treatment times was sufficient to obtain an emulsion that was stable 24 hours or more as observed by eye.

Conclusion

This experiment demonstrated that conducting a premix through a magnetic field improved the storage stability of an oil-in-water premix. The emulsions showed coagulation and phase separation in the period between 6 and 24 h, while in the premix phase separation was observed within minutes. Nevertheless, the storage stability of the emulsions was still relatively short. This could be related to the configuration of the liquid containing reservoir used in this experiment. The rectangular shape of the Haake recirculation bath probably provided stagnant zones, which may have resulted in an incomplete treatment of the premix. The presence of untreated premix, containing large micelles, in the final emulsion probably resulted in the limited storage stability of the obtained emulsion.

EXAMPLE 2: Magnetic preparation of an oil-in-water emulsion using cylindrical flask with a conical bottom as liquid-containing reservoir

<u>Research equipment and procedures</u>

1. Preparation of the premix

1,163 kg of fatty acid and 0,076253 kg of surfactant were added to 2,504 kg of de-ionised water (in a 1L-beaker). The fatty acid 'radiacid 0166' and the surfactant 'radiasurf 7403' were provided by Oleon company. This mixture was vigorously stirred with an IKA RE16 mixer at 300 rpm. The suspension was slowly heated to a temperature of 63 °C with an IKA RCT heater. The temperature was controlled with an IKA ETS-D4 fuzzy-apparatus.
When the temperature had reached 60,5 °C, 14,620 g of a 50 wt% NaOH-solution was added dropwise, while stirring of the mixture was continued. The suspension was allowed to cool to 40 °C.

2. Magnetic treatment

1,5 l of the suspension (40 °C) was poured into a liquid containing portion of 2 l. Said liquid containing portion consisted of a cylindrical flask with a conic bottom. Tubing (Masterflex Tygon lab I/P 70, Cole-Parmer Instrument Company) was attached to the bottom of this flask. Via the pump (Masterflex I/P) the emulsion was pumped to the inlet on top of the reactor. A commercial magnet (CEPI® R1/2D) was attached to the end of the tubing at the inlet of the reservoir (Fig.1B). The pump was turned on at a flow rate of 4,7 l/min (determination of flow rate performed with tap water).

3. Sampling

The emulsion was sampled at specific times (Table 2) . The samples were stored at room temperature (20-25°C) or in the refrigerator (7°C) for visual inspection. An overview of the sampling data is given in the table below. Samples s0, with a time indication 0', was taken after one pass of the emulsion through the magnet. Sampling of s0 happened on the outlet of the magnetic device, while samples s1-s3 were taken in the cylindrical storage flask.

time (min)	sample room temp	sample refrigerator
0'	s0	s0
15	s1	s1
30	s2	s2
60	s3	s3

Table 2: timing of sampling and sample notations

4. Determination of emulsion stability

The stability of the emulsions in function of the time was monitored observing by eye. Samples were collected in polystyrene cuvettes (2,5 ml) and in small glass vials (7 ml).

Results

3,5 hours after sampling, s0 samples stored in the refrigerator as well as those stored at room temperature showed phase segregation. The more intensively treated samples were still stable after 21 hours after sampling. Inspection of the s1, s2 and s3 samples after 92 hours of storage revealed some phase separation.

Conclusions

Compared to Example 1 the storage stability of the emulsion was significantly improved by using a cylindrical flask with conical bottom as liquid containing portion
Another important conclusion is that a single passage through the magnet was insufficient to obtain a stable emulsion

EXAMPLE 3: magnetic effect

Experiment 1

Research equipment and procedures

1. Preparation of the pre-mix

1,163 kg of fatty acid and 0,076253 kg of surfactant were added to 2,504 kg of de-ionised water (in a 1L-beaker). The fatty acid 'radiacid 0166' and the surfactant 'radiasurf 7403' were provided by Oleon company. This mixture was vigorously stirred with an IKA RE16 mixer at 300 rpm. The suspension was slowly heated to a temperature of 63 °C with an IKA RCT heater. The temperature was controlled with an IKA ETS-D4 fuzzy-apparatus.
When the temperature had reached 60,5 °C, 14,620 g of a 50 wt% NaOH-solution was added dropwise, while stirring of the mixture was continued. The suspension was spontaneously cooled to 40 °C. The pre-mix was stored for 5 days at room temperature. Before use, the pre-mix was stirred once more (IKA RE16 ; 300 rpm) but not heated.
One day later, the same batch of pre-mix was used for a blanc experiment after stirring with an IKA RE16 mixer at 300 rpm.

2. Magnetic treatment

250 ml of the suspension (room temperature) was poured into a cylindrical flask (2 l) with a conic bottom. A commercial magnet (CEPI® R1/2D) was mounted in a loop at the outlet of the reservoir (Fig.1C) The loop consisted of tubing of the type Masterflex Tygon lab I/P 70, Cole-Parmer Instrument Company. The tubing connects the magnet via the pump (Masterflex I/P) with the inlet on top of the reservoir. The flow rate was 4,7 l/min (measurement conducted with tap water).

The same set-up was used for the blank experiment, except for the magnetic device which was left out.

3. Sampling

After specific treatment times, the emulsion was sampled. Sampling was done at the end of the tubing positioned at the inlet of the reservoir. During sampling the pump was turned off. The samples were stored at room temperature (25 °C) and in the refrigerator (7 °C). An overview of the sampling data is given in table 3. The approximative number of passages as given in table 3 was estimated based on the volume of pre-mix introduced in the reservoir and the flow.

	time (min)	number of passages	sample room temp	sample refrigerator
experiment with magnetic device	0'	1	s0	s0
	1	12	s1	s1
	2	24	s2	s2
	15	180	s3	s3
blanc experiment	2	24	b2	b2
	15	180	b3	b3

Table 3 : timing of sampling and sample notations

4. Determination of emulsion stability

The storage stability of the emulsions was inspected by eye at different time points during storage. Samples were collected in polystyrene cuvettes (2,5 ml) and in small glass vials (7 ml).

Results

All samples prepared with the magnetic device had a certain stability, clearly improved by prolonging the treatment time. After 18 hours storage, sample s0 showed phase separation at both storage temperatures. Samples s1 and s2, in the refrigerator as well as at room temperature, were still stable after 22 h, but inspection after 42 hours revealed phase separation. Sample s3, stored at room temperature, de-mixed nine hours later, this is 51 hours after the treatment. Fourteen days after treatment, the sample s3 stored in the refrigerator was still stable.

The storage stability of the samples of the blank experiment was clearly lower. Independent of the storage temperature, samples b2 and b3 showed phase separation within 20 hours after treatment.

Conclusions

A clear difference in emulsion stability was observed between samples obtained in a system with and without a magnetic device. This demonstrates that conducting an emulsion through a magnetic field contributes to the formation of a stable emulsion.

Experiment 2

Research equipment and procedures

1. Preparation of the pre-mix

1,163 kg of fatty acid and 0,076253 kg of surfactant were added to 2,504 kg of de-ionised water (in a 1L-beaker). The fatty acid 'radiacid 0166' and the surfactant 'radiasurf 7403' were provided by Oleon company. This mixture was vigorously stirred with an IKA RE16 mixer at 300 rpm. The suspension was slowly heated to a temperature of 63 °C with an IKA RCT heater. The temperature was controlled with an IKA ETS-D4 fuzzy-apparatus.
When the temperature had reached 60,5 °C, 14,620 g of a 50 wt% NaOH-solution was added dropwise, while stirring of the mixture was continued. The suspension was allowed to cool to 40 °C. The heater was turned off and the pre-mix stored for 11 days at room temperature. Before use, the pre-mix was stirred once more (IKA RE16 ; 300 rpm) but not heated.

2. Magnetic treatment

The emulsification device used in this experiment comprised a liquid reservoir consisting of a cylindrical flask with a conical bottom. A commercial magnet (CEPI® R1/2D) was mounted in an external loop according to the scheme of Fig. 1C. The loop consisted of tubing (Masterflex Tygon lab I/P 70, Cole-Parmer Instrument Company) connecting the magnet via the pump (Masterflex I/P) with the inlet on top of the reservoir. 250 ml of the suspension (room temperature) was pumped from a beaker into the reservoir of the unit via the magnet and the tubing. The flow rate was set at 6,3 l/min (measurement conducted with tap water).

The same setup is used for the blank experiment, except for the magnetic device.

3. Sampling

After 1 hour of treatment, the emulsion was sampled in two ways. One sample was taken at the outlet of the tubing, the other in the reservoir itself. The samples were stored at room temperature (25 °C) and in the refrigerator (7 °C) for visual analysis. An overview of the sampling scheme is given in the table 4.

	sampling	sample room temp	sample refrigerator
experiment with magnetic device	reservoir	s1	s1
	tubing	s2	s2
blanc experiment	reservoir	b1	b1
	tubing	b2	b2

Table 4: sampling scheme

4. Determination of emulsion stability

The stability of the emulsions was inspected by eye. Samples were collected in polystyrene cuvettes (2,5 ml) and in small glass vials (7 ml).

Results

In the refrigerator the four samples were still stable after 4 days of storage. Upon storage at room temperature, phase separation occurred in both samples of the blank experiment (b1 and b2) in the period between 29,5 and 70 hours. Samples s1 and s2 obtained with the magnet in the loop, were much more stable and showed phase separation somewhere between 101 and 165,5 hours of storage at room temperature. Samples s1 and s2, stored in the refrigerator, were still stable after 197,5 hours of storage.

Conclusions

This experiment confirmed the contribution of the magnetic field to the stabilisation of the emulsion. Furthermore, improved storage stability was observed as compared to the previous

experiments. This improved stability can be attributed to the higher flow rate and to the fact that the emulsion was pumped into reservoir through the magnetic device.

EXAMPLE 4: Stability of an emulsion stored in a larger recipient

Research equipment and procedures

1. Preparation of the pre-mix

1,163 kg of fatty acid and 0,076253 kg of surfactant were added to 2,504 kg of de-ionised water (in a 1L-beaker). The fatty acid 'radiacid 0166' and the surfactant 'radiasurf 7403' were provided by Oleon company. This mixture was vigorously stirred with an IKA RE16 mixer at 300 rpm. The suspension was slowly heated to a temperature of 63 °C with an IKA RCT heater. The temperature was controlled with an IKA ETS-D4 fuzzy-apparatus.
When the temperature had reached 60,5 °C, 14,620 g of a 50 wt% NaOH-solution was added dropwise, while stirring of the mixture was continued. The suspension was allowed to cool to 40 °C.

2. Magnetic treatment

1,5 l of the suspension (40 °C) was poured into a 2 l cylindrical flask with a conical bottom operating as liquid reservoir. The external loop consisted of tubing (Masterflex Tygon lab I/P 70; Cole-Parmer Instrument Company). Magnet (CEPI® R1/2D) and peristaltic pump were mounted in the loop according to Fig.1C. The pumping speed was 4,7 l/min (measurement conducted with tap water).

3. Sampling
After one hour of treatment the emulsion was pumped through the magnet into a 1 l glass Erlenmeyer. Ook dit weer naar boven toe

4. Determination of emulsion stability

The stability of the emulsions was inspected by eye. The Sample was collected in a 1 litre glass Erlenmeyer.

Results

The sample stored in the Erlenmeyer flask had a storage stability of more than 19 days. The higher stability of the sample stored in the Erlenmeyer as compared to the stability of the samples stored in the 2,5 ml cuvettes and the 7 ml vials is known in the art. The surface to volume ratio, the shape and the chemical nature of the recipient can be critical, especially when stability over long storage periods is desired.
The evaluation of the emulsion stability of the samples in small flasks as done in Examples 1-3 represents an accelerated stability test.

EXAMPLE 5: Magnetic treatment of milk

The effect of magnetic treatment on the size of the lipid micelles of milk is analysed in this report. Two different types of milk were used : semi-skimmed milk and whole milk.

Research equipment and procedures

1. Milk

Two different types of milk were used in the experiments. The semi-skimmed milk, manufactured by Stassano, contains 1,6 g / 100 ml of lipids and 3,3 g / 100 ml of proteins. The whole milk, by the same manufacturer, consists of 3,6 g / 100 ml of lipids and 3,3 g / 100 ml of proteins.

2. Magnetic treatment

The emulsification device used in this experiment comprised a liquid reservoir consisting of a cylindrical flask with a conical bottom. A commercial magnet (CEPI® R1/2D) was mounted in an external loop according to the scheme of Fig. 1D. The loop consisted of tubing (Masterflex Tygon lab I/P 70, Cole-Parmer Instrument Company) connecting the magnet via the pump (Masterflex I/P) with the inlet on top of the reservoir. This entire setup is positioned in a box filled with water and ice. The magnet, part of the tubing and part of the reservoir is immersed in the water (Figure 1D). This setup allows keeping the temperature of the milk at about 20 °C.

When the milk has been poured into the reservoir, the pump is turned on at a flow rate of 4,7 l/min (measurement conducted with tap water in an identical setup).

3. Analysis

The dimensions of the fat globules are analysed with diffusive light scattering (DLS). A high performance particle sizer with a He-Ne-Laser (2,5 mW), built by the ALV-company, was used for this purpose. Measurements were performed with water as standard reference for viscosity. Correction for viscosity was not made. The dimensions of the micelles should therefore be interpreted as relative rather than absolute values.

Semi-skimmed milk

In this experiment 500 ml of semi-skimmed milk is poured into the reservoir. After 5 and 10 minutes of magnetic treatment, a sample is taken in the reservoir. The dimensions of the lipid micelles are analysed with DLS.

Three important effects of magnetic treatment are observed in Figure 2. At first an important reduction of the number of micelles sized between 5,000 and 45,000 nm is clearly shown. After 10 minutes of treatment these large micelles have completely disappeared. The second micelle size of importance is centred on 600 nm. The shape of the size distribution of this type of micelles after 5 minutes of treatment is probably caused by the disruption of larger micelles. Further treatment results in narrowing of the micelle size distribution centred around 600 nm. At last a shift of the 20 nm peak over 13 nm to 9 nm is observed upon treatment. It is very likely that these particles are individual molecules instead of micelles.

It should be stressed that no viscosity correction was made, assuming that the samples have the same viscosity as water. As a consequence the micelle sizes identified by DLS are relative instead of absolute values.

Whole milk

In this experiment 400 ml of whole milk is poured into the reservoir. After 5, 10 and 30 minutes of magnetic treatment, a sample is taken in the reservoir. The dimensions of the lipid micelles are analysed with DLS.

Similar observations as in the experiment with semi-skimmed milk can be derived from Figure 3. The number of lipid micelles with sizes between 4,000 and 53,000 nm is fiercely reduced upon

magnetic treatment. After 10 minutes of treatment these large micelles have almost en[redacted] disappeared. The disruption of said large micelles resulted in an increased presence of micelles having a particle size centred on 600 nm. As in the previous experiment shift of the smallest particles towards even smaller sizes was also observed.
It should be stressed that no viscosity correction was made, assuming that the samples have the same viscosity as water. As a consequence the micelle sizes identified by DLS are relative instead of absolute values.

Conclusions

By means of two well-known emulsions, semi-skimmed and whole milk, the influence of magnetic treatment on the sizes of the micelles was demonstrated. The micelle size distribution upon magnetic treatment was clearly changed. Recirculating milk through a CEPI® magnetic device results in a disappearance of micelles with dimensions larger than 5,000 nm. Treatment of the milk results in a uniformisation of the size of the fat globules centred on ca. 600 nm.

CLAIMS

1. A method for the preparation of an emulsion characterized in that the method comprises conducting a 'premix' of immiscible liquids through a magnetic field.
2. A method according to claim 1 whereby the 'premix' is recirculated through a magnetic field.
3. A method according to claim 1 to 2 wherein the 'premix' of immiscible liquids comprises an emulsifier.
4. A method according any of the claims 1 to 3 whereby the immiscible liquids are oil and water.
5. A method according to claim 4 for the preparation of an oil-in-water emulsion.
6. A method according to claim 4 for the preparation of a water-in-oil emulsion.
7. A method according to any of the claims 1 to 6 whereby the premix is conducted through a body with a permanent magnetic field .
8. A method according to any of the claims 1 to 7 whereby the flow rate of the premix through the magnetic field is below the flow rate of the transition from laminar to turbulent flow.
9. A method for the modification of the size of the micelles in a stable emulsion said method comprising conducting the emulsion through a magnetic field.
10. An emulsification device comprising a liquid containing device, a magnetic device and a pumping system said emulsification device allowing to recirculate a 'premix' through the magnetic device.11. Use of a device according to claim 10 for the preparation of an emulsion according to the method of any of the claims 1 to 9.

Abstract

The invention relates to a new method and related equipment for the preparation of emulsions. According to the method of the invention stable emulsions are obtained by conducting an unstable pre-mixture of immiscible fluids through a magnetic field.

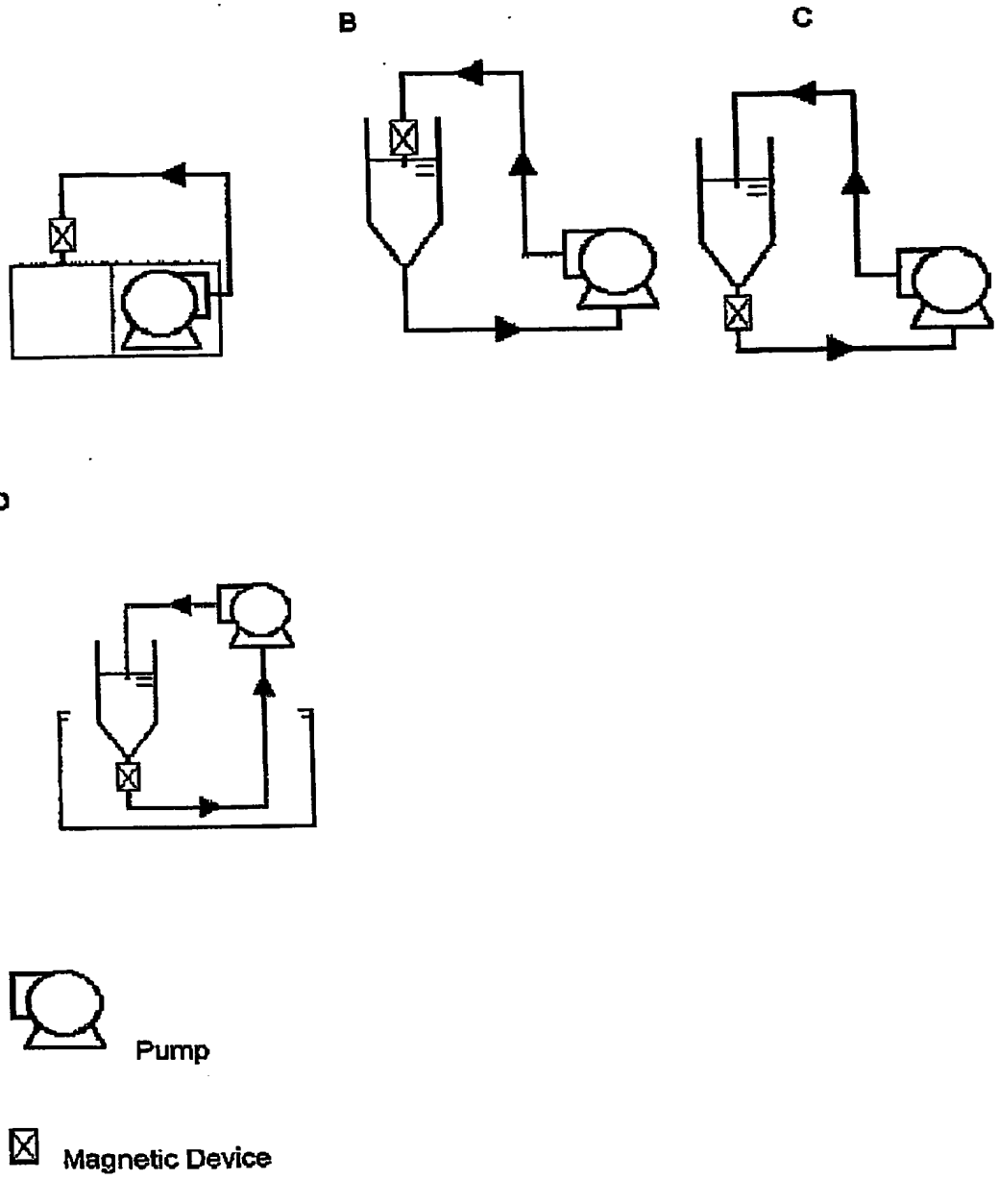

Figure 1

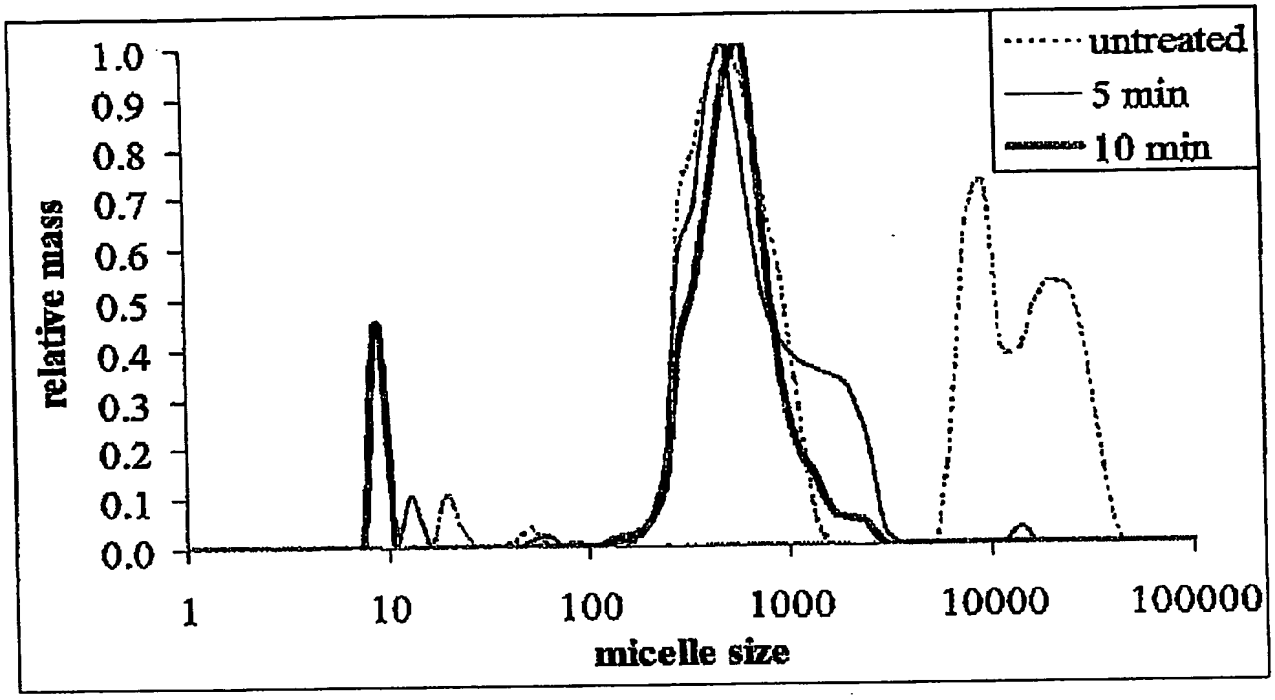

Figure 2

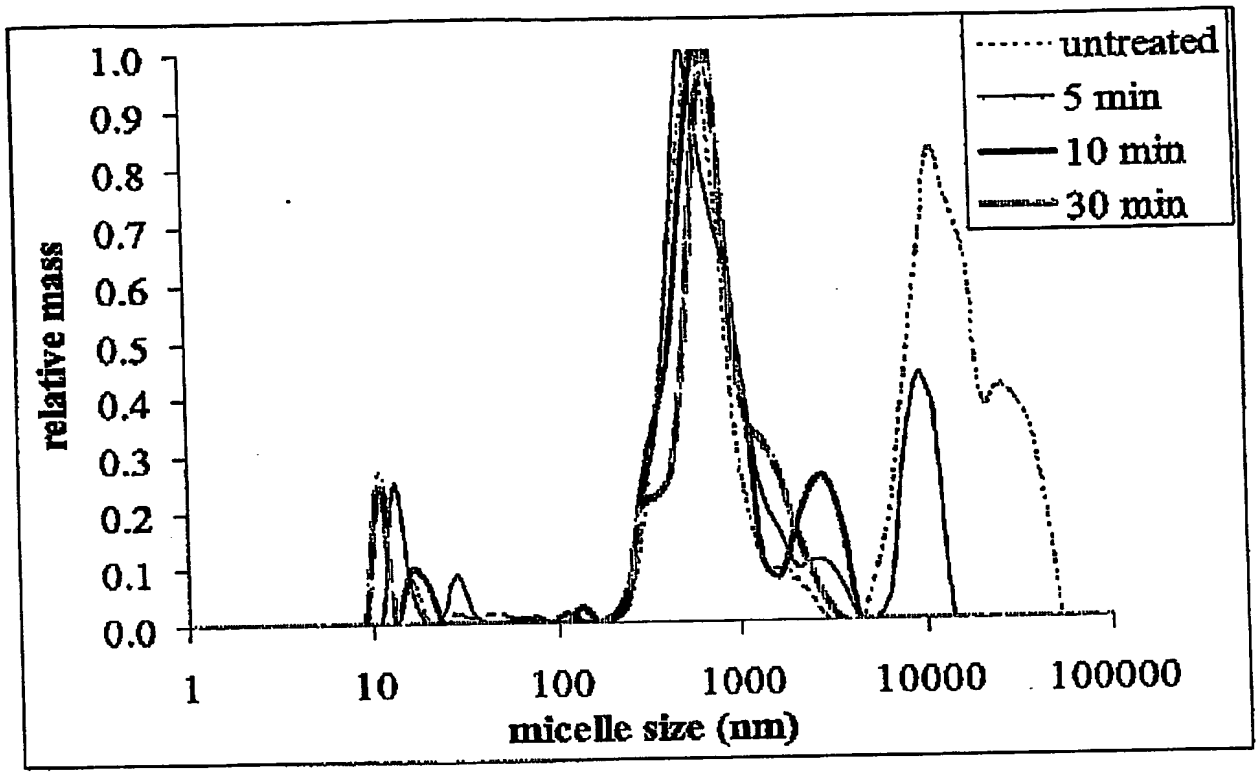

Figure 3

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